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There is a hole in my bucky

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JA951186F

J7004-m1

SUPPLEMENTARY MATERIAL FOR

There is a Hole in my Bucky

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N-MEM- ketolactam 3 from 5.6 N-MEM-azafulleroid 1

A solution of 21 mg (25.5 μ mole) 5.6 N-MEM-azafulleroid 1 in 75 ml HPLC grade 1,2-dichlorobenzene was irradiated with a flood lamp provided with a sheet of Kapton 500HN (DuPont) as the light filter, while bubbling through a gentle stream of oxygen. The temperature of the reaction was maintained at 25-30°C using a water bath, cooled internally by tap water running through a copper spiral. After 3 hours, the whole reaction mixture was poured on a column of Silica/toluene (25 cm). Upon elution with toluene, a trace of starting material and the 1,2-dichlorobenzene were removed. Elution with toluene/EtOAc (9:1) gave the fraction containing pure ketolactam. The solvent was removed *in vacuo*, and the residue was dissolved in a minimum amount of CS₂. The product was precipitated with Et₂O (50 ml), the mixture was centrifuged, and the supernatant was decanted. The solid residue was washed with Et₂O (2 x 70 ml; suspended, centrifuged, decanted) and dried in a vacuum oven at 60°C overnight. Yield: 14 mg (64%).

J7004-m2

^1H NMR (CS_2 , TMS; 500 MHz, δ): 6.19, 5.83 (AB system; $J_{\text{AB}} = 10.5$ Hz; 2H; N- CH_2), 3.89 (m, 2H; O- CH_2CH_2), 3.48 (m, 2H; O- CH_2CH_2), 3.23 (s, 3H; OCH_3).

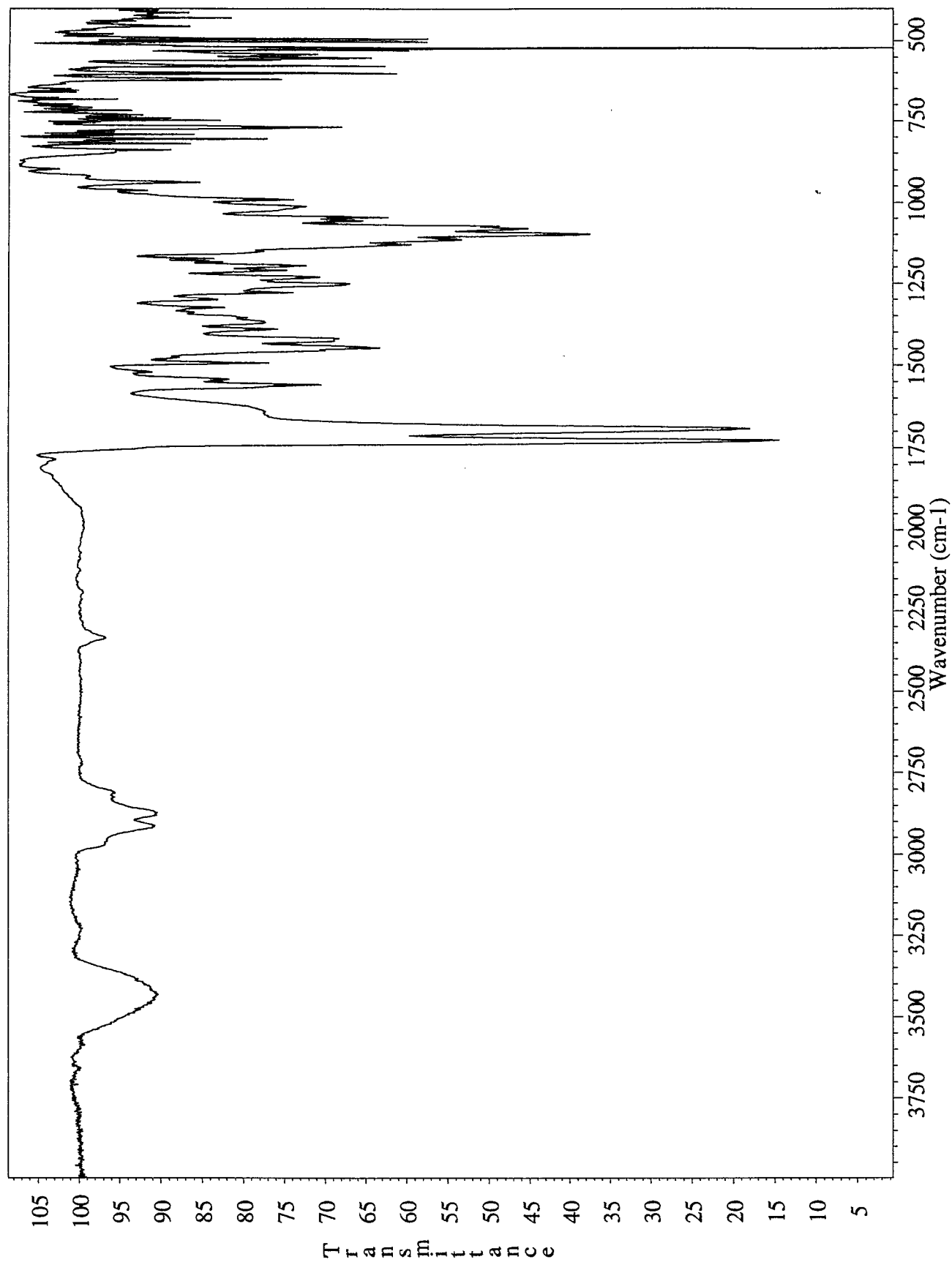
^{13}C NMR (CDCl_3 , TMS; 125 MHz): 198.49, 163.58, 150.11, 149.40, 147.75, 147.43, 147.11, 146.74, 146.63, 146.43, 146.25, 146.13, 146.08, 145.93, 145.81, 145.46, 145.40, 145.34, 145.16, 144.91, 144.84, 144.55, 144.45, 144.25, 144.16, 143.99, 143.84, 143.78, 143.63, 143.52, 143.43, 142.69, 141.63, 141.22, 141.05, 140.41, 139.75, 139.61, 139.49, 139.28, 138.72, 137.93, 136.72, 136.03, 135.96, 135.71, 134.05, 133.96, 132.99, 132.43, 132.05, 128.23, 127.96, 80.61, 71.64, 70.04, 59.11.

FTIR (KBr): 1727 s, 1693 s, 1083 s, 1075 s, 619 m, 600 m, 578 m, 559 w, 554 m, 552 m, 541 w, 528 m, 522 s, 503 m, 494 m, 458 m (cm^{-1}). UV-vis (CHCl_3) λ_{max} (nm):

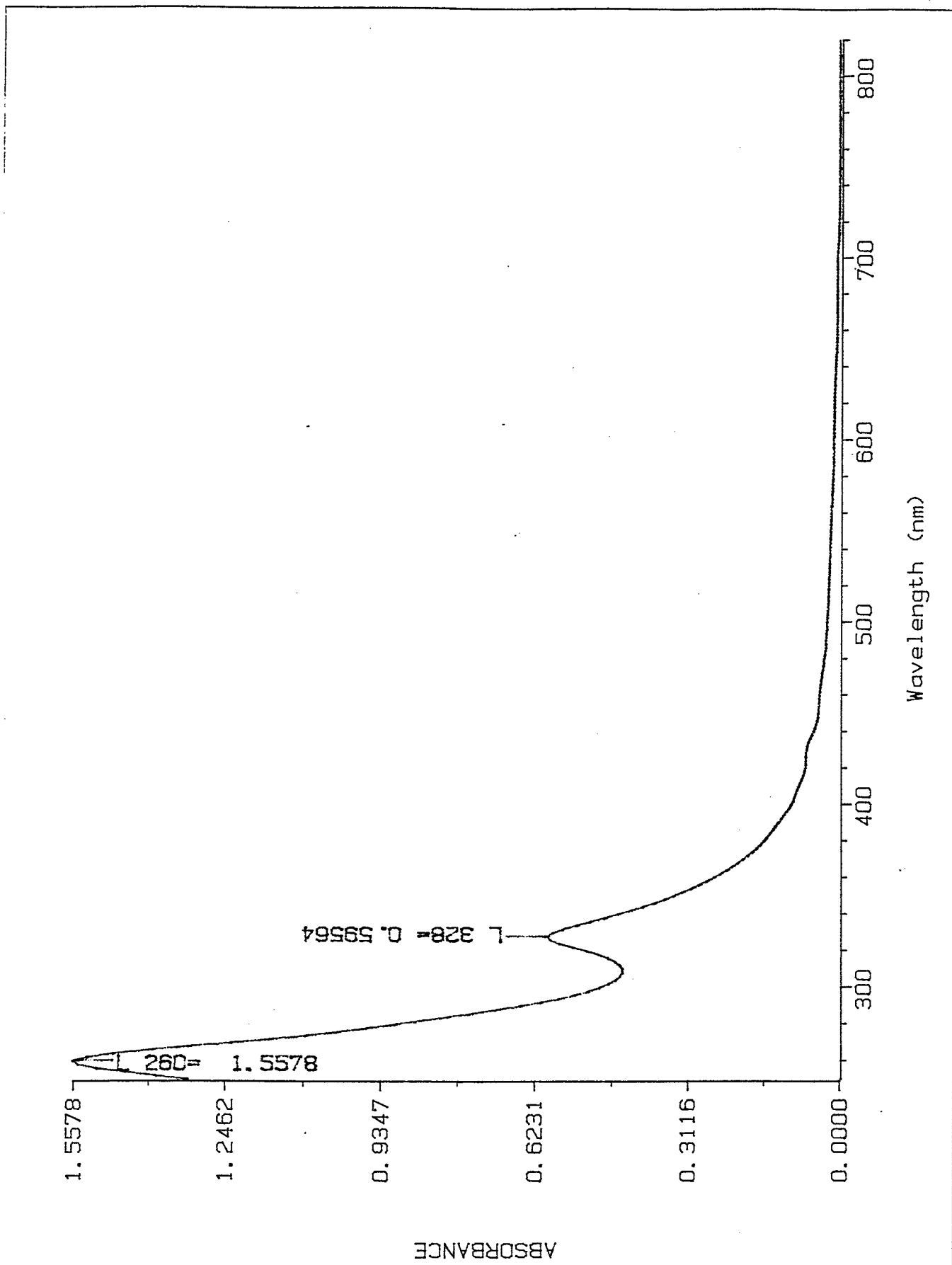
260, 328, 428 (sh), 466 (sh), 616 (sh), 688. FABMS (NBA) m/z : 856 ($\text{M}+\text{H}$) $^+$. Anal. Calcd for $\text{C}_{64}\text{H}_9\text{NO}_4$: C, 89.82; H, 1.06; N, 1.64. Found: C, 87.21; H, 1.32; N, 1.61.

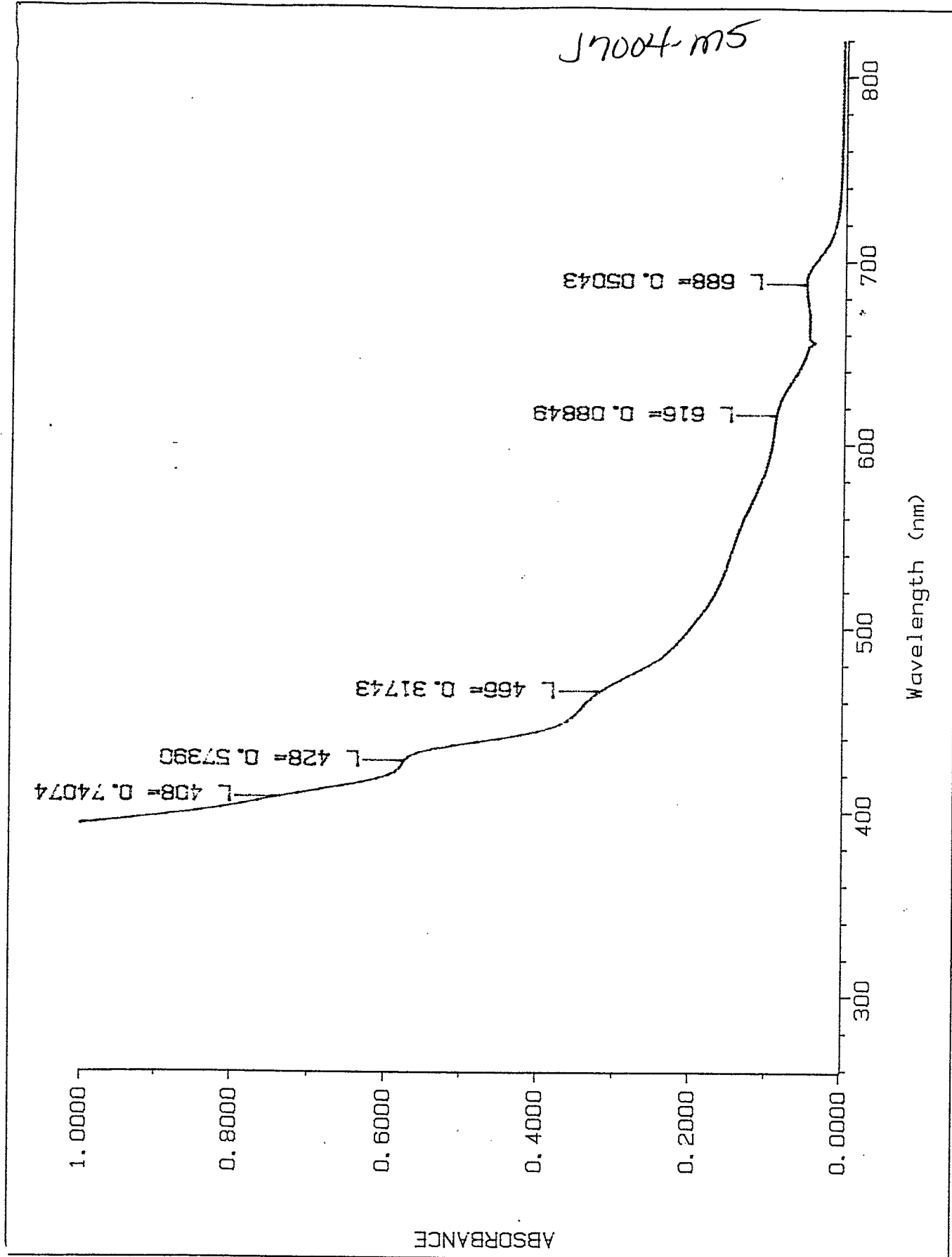
J7004-m3

MEM Ketolactam

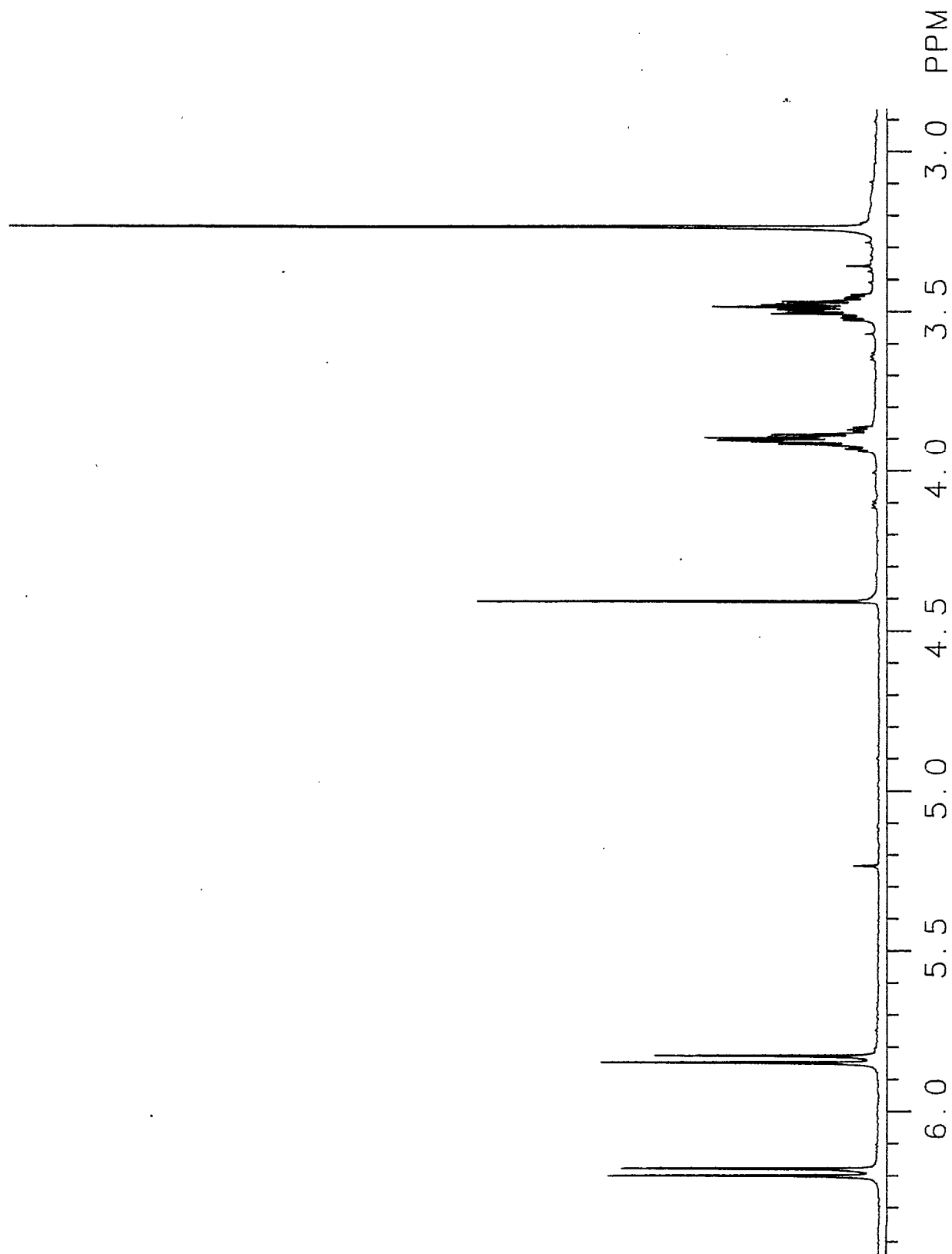


J7004-m4



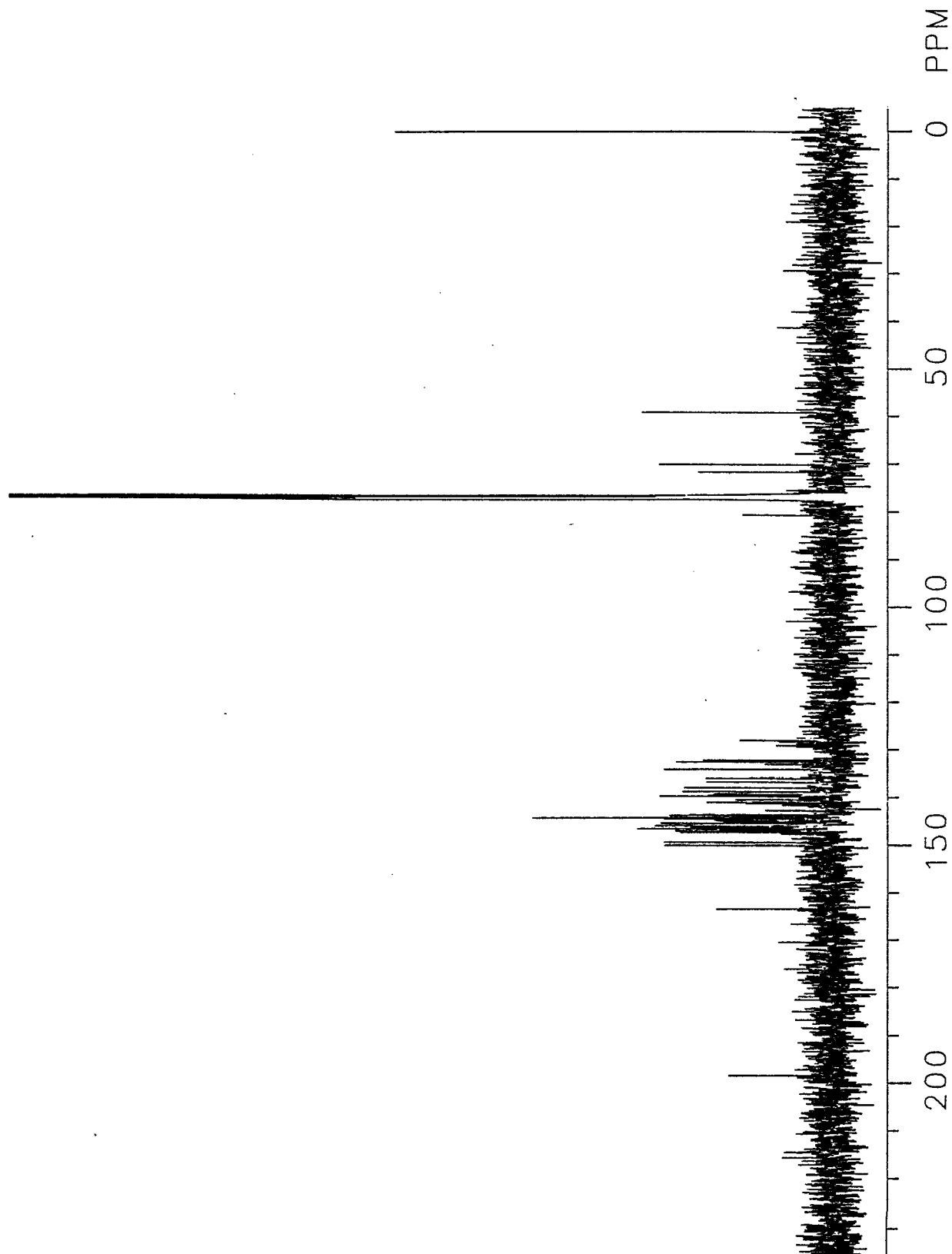


J7004-m6



J7004-m7

1-15-2-95



J7004-m8

2-15-2-95

